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COLD PLASMAS:

I. ELECTRICAL CONDUCTIVITIES OF SODIUM-AMMONIA SOLUTIONS

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<u>Cold Plasmas.</u>

I.   ELECTRICAL CONDUCTIVITIES OF SODIUM-AMMONIA SOLUTIONS*

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ABSTRACT

Electrical conductivities of sodium ammonia solutions have been measured from -80 to +185°C, covering the complete liquid range and in the case of dilute solutions, some of the gaseous range. The objective of these measurements is the evaluation of these solutions as media for gaseous plasmas studies. The attractive features of these solutions are long lifetimes (the plasma being at equilibrium), temperatures low as 133°C, and high electrical conductivities.

Visual appearances of the dense gases are the same as those of the liquid; gases with low concentrations of sodium being blue and ones with high concentrations having yellow-red metallic copper-gold appearances.

In the experimental data for the liquids, conductivity maxima occur above 80°C. In the case of dilute solutions, the maxima temperatures are close to the critical temperatures. Because of this, conductivities of supercritical gases have been measured which are higher than the corresponding liquid conductivities at room temperature.

1. INTRODUCTION

The objective of this program is to prepare compressible gaseous plasmas under equilibrium with high electrical conductivities. Dense gases of metals satisfy these requirements. Birch [1] obtained conductivities in dense mercury gases (1400°C at 2700 kg/cm^2) as high as 10% of that of liquid mercury at room temperature. Such dense gases are not attractive for plasma studies, however, because experimentation under these extreme conditions is difficult. Therefore, the use of dense gaseous solvents to dissolve metals is being investigated. Ammonia was chosen as the gaseous solvent and sodium as the solute.

The feasibility of the program was based on the following:

(1) The known properties of dense gaseous electrolytic solutions are similar to those of liquid solutions;

(2) concentrated sodium ammonia liquid solutions have metallic characteristics; and

(3) Hannay and Hogarth [2] showed that sodium ammonia gaseous solutions have lifetimes of at least a few seconds with respect to the self-reaction between solvent and metal

$$Na + NH_3 = NaNH_2 + 1/2H^2$$

that destroys the desired electrical properties.

The first problem attacked in this program was that of sodium ammonia solution lifetimes in the gaseous state. These lifetimes are potentially long, in contrast to those of most laboratory gaseous plasmas, because the plasma is under equilibrium. The only limitation on the plasma lifetime is the chemical self-reaction. In addition, the visual appearance of the concentrated gases indicates that these gases may be metallic in nature.

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In the initial phases of the program, special attention was devoted to the problem of removing causes of the self-reaction between solvent and metal which limits solution lifetime. Highly purified solutions were sealed in glass and heated to 130-185°C. When a lifetime of 52 minutes in the gaseous state was attained, emphasis was switched to other problems. It is probable, however, that the lifetime could be extended by at least an order of magnitude. These relatively long lifetimes of these gaseous solutions mean that long useful working times are available for carrying out plasma experiments under equilibrium conditions.

Once it was established that useful lifetimes could be obtained, measurement of the electrical conductivities in both the liquid and gaseous states were begun since electrical conductivities are one of the most important of plasma parameters.

The most extensive sets of conductivity data in the literature are those for the Na-NH$_3$ solutions, there being data also for Li-NH$_3$, K-NH$_3$, Na-K-NH$_3$ and Li-CH$_3$NH$_2$ solutions. Cady[3] found that the conductances of sodium ammonia solutions are considerably greater than those of electrolytic solutions of typical salts. Franklin and Kraus[4] obtained additional conductivity data for Na-NH$_3$ solutions at the boiling point of ammonia. Kraus[5] made a definitive series of measurements of the specific conductances of solutions of sodium, potassium, lithium, and sodium-potassium in liquid ammonia. Kraus and Lucasse[6] measured the specific conductances of concentrated sodium and potassium ammonia solutions at -33.5°C. More recently, Evers, Young II and Panson[7] and Berns, Evers, and Frank, Jr.[8] measured the conductance of lithium in methylamine as a function of concentration at low temperatures.

The state of the measurements on temperature coefficients of electrical conductivities is not as complete as on the conductivity-concentrations themselves. Franklin and Kraus[4][9] made preliminary experimental observations on the temperature coefficients of sodium, Li and K in ammonia below -33°C.

Kraus[5] gave the results of two runs at temperatures up to $+85^{\circ}$C, in which the temperature coefficient of solutions of sodium in liquid ammonia was measured (the concentrations of these solutions were not reported). They were not able to reduce the rate of the self-reaction between sodium and ammonia sufficiently to get reproducible measurements. Lucasse[10] reported a few additional measurements, then Kraus and Lucasse[11] made an extended set of quantitative reproducible measurements below the boiling point of ammonia.

Panson and Evers[12] made a comprehensive set of temperature coefficient measurements on lithium in methylamine from -70° to $+10^{\circ}$C. He reported appreciable self-reaction above -20°C.

The conductivity measuring techniques used in all the reported investigations have been based on direct measurements. Unfortunately, metal electrodes are needed for these methods of measuring conductivities and these electrodes catalyze the self-reaction between solvent and solute, the reaction becoming more vigorous as the temperature is increased. Kraus[5] tried substituting metals such as gold for platinum. Obtaining no appreciable improvement, he then reduced the surface area of the electrodes to a minimum and stirred the solutions during the course of the measurements in order to make the solution more uniform and to remove bubbles produced by the self-reaction.

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2. EXPERIMENTAL APPROACH

These experimental investigations have been centered around
solution lifetimes and conductivity measurements at elevated
temperatures. At the time that this program was initiated, the
available facts were as follows. With respect to gaseous solu-
tion lifetimes, Hannay and Hogarth[2] prepared one gaseous sodium
ammonia solution, which lasted for a few seconds in the gaseous
state. This short lifetime was due to the catalysis of the
self-reaction between solvent and solute by impurities in the
solution and as well as on the walls of the container. Hence.
it was necessary for us to prepare the solutions in states of
high purity, and to use measuring techniques that would not
require contact of foreign materials with the solutions.

2.1 SAMPLE PREPARATION

Current ultra high vacuum philosophy has been adapted to puri-
fication and sample preparation even though the operating pressures
are about 100 mm. These systems are thoroughly baked out under
high vacuum to remove volatilizable contaminants. Two types of
valves are used inside the oven, both of which are bakeable,
all-glass (without stopcock grease) and non-leaking with respect
to the outside atmosphere.

Valve functions are satisfied by use of breakseals or by one of
two types of all-glass valves. Closing by use of seal-offs is
undesirable because of the contaminants evolved under the intense
heat required for seal-off. Hence, use of seal-offs has been
restricted to the one case where no other method can be used,
namely for sealing off the conductivity cells containing the
purified solutions. Each seal-off region is isolated from the
rest of the sample system during the seal-off and permanently
thereafter. The purpose of this isolation is to prevent con-
tamination of the companion by the seal-off products. In
practice, each set of two conductivity cells is serviced by its
own condensor which is connected to the ammonia purification
system by a freeze valve; there being a bank of five such units.
Thus, if excessive contamination is produced during seal-off of
one cell, this will affect, at most, a second cell.

When a leak-free closure is needed, the freeze valve is used.
This valve is in the form of a capillary U-tube. When closure
is desired, ammonia is condensed in the U-tube with liquid nitrogen.
This solid ammonia plug can be pumped to 10^{-5} mm. On several
occassions, conductivity cells have been accidently snapped
and the sudden opening to the atmosphere did not produce a
detectable change on a discharge gauge on the other side of the
freeze valve.

For less critical applications, a ground glass check valve, manipulated with an external magnet operating on an iron rod sealed inside of glass tubing, is used. This valve can be pumped to 10^{-3} mm with the other end at atmospheric pressure.

The sodium used for sample preparation and the reflux columns is prepared by triply distilling commercial sodium bars. One source was found to contain a liquid contaminant. Kern Chemical Company, 99.99% sodium, appears to have adequate purity, but is more difficult to handle. During the distillation and encapsulation in tubes with breakseals, cracking of the glass occurs frequently.

Sodium has also been prepared from multiply-recrystallized sodium azide, which is decomposed into sodium and nitrogen on heating in vacuum. The sodium so prepared was an excellent catalyst, solutions prepared with it decomposing at dry ice temperatures. This catalytic behavior disappeared after multiple distillation.

In the ammonia purification process, tank ammonia, Matheson Company, Inc., is condensed (outside of the oven) over metallic sodium and allowed to reflux. Further purification is completed inside a bakeable vacuum system (figure 1). Outside the oven, standard components such as greased glass stopcocks, Viton-A glass vacuum needle valves and liquid nitrogen traps are used. Inside the oven, glass is the only material that can come in contact with the sodium and/or ammonia in the liquid or gaseous states. After refluxing, the central fraction of the ammonia is sent into a bakeable vacuum fractionating column inside the oven. After refluxing and fractionation, the central fraction is passed into a second fractionating column. After refluxing and rejection of the initial fraction, the ammonia is used to prepare the samples. At this point, a breakseal is opened, connecting the ammonia purification system with the sample preparation system, which is adjacent to it inside the oven.

The freeze valves connecting the ammonia purification system with the sample preparation system are then cooled with liquid nitrogen.

A breakseal between the ammonia purification and the freeze valve is now broken, ammonia condenses in the freeze valves, thereby isolating each pair of conductivity cells from the others. Encapsulated sodium located between condensers and conductivity cells, is then distilled onto the walls under pumping. After this, one freeze valve at a time is opened, ammonia liquified using the condenser above the pair of cells, and the condensed ammonia then washes the sodium into the conductivity cells. The lower part of the cell is immersed in liquid nitrogen, the freeze valve refrozen, this section pumped to 10^{-5} mm, sealed off, the seal-off annealed, and the cell stored in dry ice.

2.2 CONTAINMENT

Glass vessels have been used successfully to contain hot gases
under temperatures and pressures more extreme than those required
for the containment of sodium ammonia solutions; for example,
Kistiakowsky[13] used quartz tubes to contain pressures of about
300 atmospheres at 300°C. In contrast to the usually satisfactory
performance of glass, the behavior of glass for the containment
of hot dense sodium ammonia fluids has not been satisfactory.
The rupture of the glass cells may be due to strain in the glass,
surface microcracks, or chemical attack by the hot dense sodium
ammonia solutions.

The usual cause of failure of glass tubes containing dense gases
is inadequate annealing of the glass. This source of strain was
ruled out as the prime cause of the difficulty in containing
hot sodium ammonia solutions not only by use of careful annealing,
but by use of quartz, in which the strain problem is less sig-
nificant than with pyrex. Substitution of quartz for well
annealed pyrex did not lead to any improvement.

Another source of strain, inhomogeneity of glass in the seal-
off region, was the cause of many failures. The inhomogeneity
arose from the approach used for preparing the samples. Sodium,
dissolved in purified liquid ammonia, is run into the cell.
Frequently, there was sufficient sodium deposited on the walls
in the seal-off region to react with the glass, weakening it
seriously. This problem was solved by bake-out of the cells
under vacuum before filling with solution. With this treatment,
the solutions flow down the walls without leaving appreciable
amounts of deposits. There has been no evidence of weakening
of the glass in the seal-off region since bake-out has been
introduced.

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A third source of strain is the presence of internal pressures
of hundreds of atmospheres in the gas phase, which sets up stresses
in the glass. Those stresses weaken the glass, since glass under
tension is mechanically weak. Since glass under compression is
strong, external pressurization has been introduced; however,
it did not provide a complete solution to the rupture problem.

Another effect of strain is that chemical attack is often greater
in the regions of strain than in other portions of the glass.
Thus, Holley, Neff, Weiler and Winslow[14] report than pyrex
exposed to cesium vapor at $400^{\circ}C$ for 240 hours is badly attacked
at the strain points. In addition to the strain points them-
selves, the regions where glass has been blown appear to be more
suseptible to attack. Thus, vycor, after one hundred hours at
$400^{\circ}C$, is fractured due to attack at the joints; and aluminum
silicate glass, after fifty hours at this temperature, is found
to be slightly oxidized at the joints. Thus, it is likely that
the worst feature of the strain in glasses is not the mechanical
weakness of the glass, but the reduction in ability to resist
chemical attack.

The following glasses are under evaluation for usability as cell
materials at elevated temperatures.

> Uranium Glass.   .   Corning Code No. 3320
> Aluminosilicate   Corning Code No. 1723
> Kovar Sealing Glass   Corning Code No. 7052

So far, the behavior of quartz has been similar to pyrex. One
glass, Corning No. 7052, has exhibited relatively poor strength.
Lifetime of the solutions in the different glasses will also be
measured

Another factor influencing the strength of the glass is micro-cracks on the surface, these cracks acting as stress multi- pliers[15][16][17]. The strength of glass may be increased by as much as a factor of 10 by using a hydrofluoric acid wash, which removes the surface layer. This treatment has the additional benefit that it removes sorbed surface layers of alkali hydrates, which forms on exposure of glass to the atmosphere, removed, this layer being a source of prolonged outgasing during bake-out under pumping. No increase in rupture temperature was obtained when the hydrofluoric acid wash technique was tried.

The general conclusion is that chemical attack of the hot solutions may be the prime source of the containment difficulty. It has been found that with concentrated solutions rupture occurs at lower temperatures than with dilute ones. Use of external pressurization mitigates, but does not entirely solve the rupture problem. The complete external pressurization assembly is shown in figure 3.

2.3 ELECTRICAL CONDUCTIVITY MEASURING SYSTEM

The conductivity measuring system was designed so that glass would be the only foreign material in contact with the solutions. Two types of inductive electrical conductivity measurement techniques were evaluated. One was based on measurements of solutions contained in straight lengths of glass tubing, and the second, in toroids (figure 2). From the viewpoint of accuracy, the toroidal technique proved to be superior and was adopted.

The sodium ammonia solutions are contained in conductivity cells with a toroidal section. Two ferrite toruses are linked through the solution torus. Coils are wound about each ferrite torus. An input signal is sent into one coil, current being induced in the ferrite coil, the solution torus, the second ferrite core, and out through the second coil, the output being measured at the instrument panel. An internal heater is located parallel to, but below the conductivity cell, an external heater being placed around the bomb. The complete assembly inside the bomb is shown in figure 4.

The transfer function across the system is dependent, not only on the geometry and conductivity of the sample, but also on the frequency of the signal. For this reason, in operation, the approximate conductivity of the solution torus is determined and the correct frequency used for that sample.

The conductivity system is calibrated with toruses fabricated accurately either with liquid mercury or solid resistive materials. The transfer functions for the liquid and solid metals are in complete agreement with each other.

3. VALIDATION OF EXPERIMENTAL DATA

There are many experimental details that make the evaluation and interpretation of the data difficult. In particular, it is necessary to detect when spurious effects are occurring and to establish the limits of accuracy attainable in the absence of spurious effects.

The principal spurious effects are instrumental changes, decompositions of solutions, and occasionally, erratic behavior near the critical point.

The conductivity cell is placed inside a high pressure bomb before raising the temperature. Windows have not been installed so that it is not possible to make visual observations. In the earlier experiments, the conductivity cells were fabricated from heavy walled pyrex capillary and tested behind glass barricades. It was found that visually observable effects occurred at the same time as some of the peculiarities in the conductivity measurements. Vigorous turbulence was often observed just below the critical point; at the same time, the conductivity data became erratic, varying by as much as a factor of ten over a period of a few minutes.

Bubbles in the capillary were associated with sudden increases in resistance. This problem was eliminated for dilute solutions when larger diameter tubing was substituted for capillary, although the problem may still be present with the concentrated solutions, since these are very viscous.

Because of the corrosiveness of the solutions, there have been many difficulties with instrumental changes. When cells at temperatures of 150 to 200°C rupture, the sodium ammonia solutions react with the instrumental wiring in the interior ·

of the bomb. The wiring is changed whenever check-outs prove
unsatisfactory. Since failures occassionally occur during runs,
a system was installed for calibrating the conductivity measuring
fixture at high temperatures and pressures while the run is taking
place.

This external calibrator consists of a wire torus parallel to
the conductivity cell torus which is open at one point. Two
leads connect this to a switch. When the switch is open, the
torus is open; when the switch is closed, the torus circuit is
closed through an external resistor. The external calibrator is
treated a a secondary standard. This unit is calibrated as a
complete torus.

3.1 CONDUCTIVITY MEASURING TECHNIQUES

The purpose of the reduction of the data is to determine the specific conductivity of each solution, σ, as a function of the temperature, t, and the mole function of Na, X_2. The steps in the process for measuring σ are:

(i) calibration of conductivity cell

(ii) calibration of the conductivity fixture

(iii) measurement of the solution resistances.

The conductivity measuring system is calibrated with toruses made from solid materials (secondary standards) and from liquid mercury. Use of liquid mercury toruses provides additional confidence in the general conductivity measuring technique since mercury, like the Na-NH$_3$ solutions, is a liquid conductor. In addition, the mercury toruses are carefully constructed, calibrated and used as primary standards. The data for the solid resistors agreed quantitatively with those for the mercury toruses.

The purpose of calibrating the conductivity cell is to establish the ratio C, of the resistance of a solution in the cell torus to its resistivity. The following techniques are used to calibrate the conductivity cells:

(i) Geometrical
The total length of the torus is divided by the cross-sectional area of the glass tubing used by the glassblower. This method is useful for establishing a rough value of the cell constant

(ii) Direct Current Plunger
A plunger is inserted into the cell, blocking electrical continuity in the torus. The blocked torus is filled with mercury and the direct current conductivity measured. After correcting for the presence of the plunger, the cell constant

is the ratio of the corrected resistance to the resistivity of mercury.

(iii) Inductive Method Using Liquid Mercury
The cell is filled with mercury and the resistance measured inductively. The cell constant is the ratio of this resistance to the resistivity of mercury.

A number of operational procedures have been used to calibrate the conductivity fixture and to measure the solution resistances. In the calibration procedure, known toroidal resistors are placed between the two ferrite cores, and the transfer functions measured. Then a conductivity cell is placed in the fixture, and the transfer functions measured again. The ratio of transfer functions of cell to standard are equal to the ratios of their resistances.

At first, a graphical technique has been used. The calibration establishes a curve of V^O (output voltage) to toroidal resistance. Next, the fact that $\frac{RV^O}{V^i}$ is constant was used (the V^O technique). More recently, the external calibrator has been used. This places a known resistance in parallel with that of the solution resistance. In addition, a figure-eight external torus has been used such that the current it transfers is in opposition to that transferred by the cell. A minimum or null output results when the two resistances become equal.

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3.2 REPRODUCIBILITY OF THE DATA

In more recent runs, samples have been heated and cooled to evaluate whether or not measurable decomposition has taken place
Each of the two runs, shown in figure 5, were heated three separate times. The reproducibility of the data shows that the experimental techniques are valid.

The heating system is designed to obtain small temperature drops rapidly when desired. Thus, when there is a sudden change of conductivity, the temperature can be dropped to see whether the conductivity data at lower temperatures are reproduced.

4. EXPERIMENTAL RESULTS

The first experimental aspects of the program were concerned
with establishing that appreciable lifetimes of the gaseous solu-
tions could be obtained (with respect to the self-reaction of metal
with solvent) in the gaseous states. As experimental purification
techniques were improved, the lifetimes were increased to 52
minutes at $145^{\circ}C$ in the gas phase.

Next, the possibility of placing materials, other than glasses,
in contact with the solutions was investigated. So far, the only
non-catalytic structural material reported in the literature is
glass. Organo-silane coatings, prepared from Beckman Desicote
18772, were prepared.

Substituted chlorosilenes and alkoxy-silanes are readily deposited
on glass and other materials.[18] The action is a hydrolytic
one, in which adsorbed water on the surface reacts with the
silane, liberating hydrogen chloride from a chlorosilane or
ethanol from an ethoxy silane and leaving a film of polymerized
substituted siloxane integrally attached to the surface. The
polar Si-O bonds apparently exhibit an infinity for the similarly
constitute surface structure of the glass surface and the organic
radicals are directed outward. Since the alkali metals are fre-
quently stored under hydrocarbons and related compounds, it
appeared possible that a surface with a hydrocarbon exterior
might provide an inert non-catalytic surface for the sodium
ammonia solutions.

E. P. Arthur[18] found that a hydrolyzed silane layer did not
impair the pH response of glass electrodes or appreciably in-
crease their resistance.

When silane coated materials were placed in test cells containing
sodium ammonia and the cells heated, vigorous bubbling appeared
in the region of the silane surfaces. The solution in this

region seemed to decompose more rapidly than elsewhere in the cell; for this reason tests were not continued at that time.

4.1 ELECTRICAL CONDUCTIVITIES

The function of the low temperature data has been primarily to overlap our conductivity data with those of Kraus[5] and Lucasse[6], our temperature coefficient data with those of Kraus and Lucasse.[11] Our concentrations are deduced by comparing our conductivities at the boiling point of ammonia with their concentration conductivity correlation. Although a gas analyzer has been installed to measure chemical compositions of the samples directly, too many samples have ruptured at elevated temperatures, removing the possibility of future analyses. All concentrations reported herein have been obtained using the above conductivity concentration.

The low temperature data are plotted in figure 6 and summarized in Table I. It is seen that the temperature coefficients generally agree with those of Kraus and Lucasse.[11]

Above room temperature, the conductivities pass through maxima above $80^{\circ}C$ in the case of dilute solutions, the maxima flattening into plateaus at higher concentrations (figure 7 and Table II). In addition, the maxima appear to be at higher temperatures at higher concentrations.

At the highest temperatures, the fractional filling of the cell influences the behavior strongly. If the fractional filling is too large, so that the density exceeds the critical density, then the cell is filled with liquid below the critical point. Several examples are shown in figure 7. The rising liquid fills the gas region (which is also shown). When this happens, the solution density remains constant with increasing temperature and the conductivity rises instead of falling.

In figure 8, the filling was correct. The peculiarity in the gas phase below the critical point is due to the torus becoming

partially filled with liquid. The temperature at which gas and liquid phases became indistinguishable, 134°C, is slightly greater than that of the pure solvent itself, 132.9°C. The gaseous conductivity then remained constant with increasing temperature.

Vigorous turbulence has been observed visually in the liquid solutions as much as 30° below the critical point when the experiments have been done in capillaries. At the same time, ther are random changes in the measured conductivities.

The data for six samples, in which such effects were present, are shown in figure 9. In other runs, such as in figure 8, the critical state was reached without any turbulence. It is possible that the turbulence is more pronounced when the densities are below the critical densities.

In the case of the more concentrated solutions, gaseous conductivities did not become measureable at temperatures of 185°C. At 80 to 100°C, the conductivities appear to plateau, then increase with increasing temperature (figure 10). It has not yet been established whether this effect is real or is due to subcritical fluctuations of the liquid state.

5. DISCUSSION

The occurence of the conductivity maxima at 80 to $100^{\circ}C$ is most fortunate because of the proximity to the critical points of dilute solutions. Because of this closeness, there are relatively small drops in conductivity, in going from the maxima to the critical points.

In order to determine the reason for the occurrance of the maxima at high temperatures, experiments are under way to measure solution densities. Techniques for measuring viscosities, electron concentrations and electron densities from $-8?$ to $+180^{\circ}C$ are under development. It is possible that the increase in conductivity with increasing temperature is due to the shift in equilibrium between dimers, e_2^- or Na_2 electrons. The maxima might even correspond to complete dissociation of the dimer.

The use of sodium ammonia gases ad media for plasma studies is promising, gaseous conductivities of 1 to 100 ohm^{-1} cm^{-1} having already been measured. Attempts are continuing to reach the critical points of more concentrated solutions.

TABLE I

LOW TEMPERATURE DATA*

Sample No.	Mole Fraction of Na at -33.5°C	Cond. Cell** Calib. Methods	Resistance*** Measuring Method	Frequency kc
3-26-18	1.145×10^{-1}	G	Vo	5
3-26-15	8.00×10^{-2}	G	Vo	7
3-26-10	5.27×10^{-2}	G	E	7
4-26-8	5.1×10^{-2}	P	E	4
10-3-1A	5.00×10^{-2}	G	Vo	10
3-26-12	4.58×10^{-2}	G	E	7
3-26-9	3.87×10^{-2}	G	Vo	10
4-26-2	3.57×10^{-2}	P	E	20
10-8-4	2.95×10^{-2}	G	E	1250
11-9-1A	2.9×10^{-2}	G	G	125
4-26-1	2.452×10^{-2}	P	E	80
10-30-3A	2.258×10^{-2}	G	G	200
3-26-16	1.875×10^{-2}	G	Vo	200
4-26-7	1.655×10^{-2}	P	E	400
4-26-5A	1.635×10^{-2}	P	Vo	400
4-26-3	1.32×10^{-2}	P	Vo	800
9-5-4A	1.175×10^{-2}	G	E	1250
2-36-17	1.075×10^{-2}	G	Vo	900
10-3-3	6.6×10^{-3}	G	E	1250
X-15	5.42×10^{-3}	G	G	125
10-8-4A	2.93×10^{-3}	G	E	1250
10-8-3	1.7×10^{-3}	G	E	1250

* All cells are made of pyrex except 11-9-1A, which is fabricated from uranium glass.

** Conductivity cell calibration methods : G-Geometrical, P-D. C. Plunger

*** Resistance Measuring Methods:

k-1 E - External Calibrator

Vo Vo - Constant transfer function $\frac{RV^o}{\nu V^i}$ where R is the cell resistance, ν the operating frequency and V^o, V^i the output and input voltages respectively.

R-Vo G - Graphical (R vs. Vo at fixed frequency)

TABLE II

CONDUCTIVITY MAXIMA*

Sample No.	Mole Fraction of Na at -33.5°C	Resistance** Measuring Method	Conductive Maxima	
			Temperature °C	ohm^{-1} cm^{-1}
3-26-15	8.00×10^{-2}	E		
3-26-12	4.58×10^{-2}	E		
9-5-3A	3.7×10^{-2}	G	109	2.1×10^2
8-22-2A	2.39×10^{-2}	Vo	111	1.7×10^2
11-9-1A	2.9×10^{-2}	Vo	86	1.4×10^2
10-30-4	2.7×10^{-2}	G	81	9.9×10
10-3-4	2.55×10^{-2}	G	81	6.5×10
11-9-3	2.5×10^{-2}	Vo	94	6.5×10
10-30-3A	2.258×10^{-2}	G	95	3.8×10
10-3-2	2.05×10^{-2}	Vo	95	3.7×10
10-30-2A	1.96×10^{-2}	E	88	2.8×10
11-9-4	1.94×10^{-2}	Vo	94	2.3×10
10-3-3A	1.82×10^{-2}	Vo	97	1.9×10
10-3-2A	1.75×10^{-2}	Vo	93	1.5×10
10-3-5A	1.72×10^{-2}	G	98	1.4×10
3-26-14	1.35×10^{-2}	E	98	7.0
9-5-4A	1.175×10^{-2}	E	93	4.1
W-1	1.178×10^{-2}	G	92	3.5
10-30-3	7.0×10^{-3}	Vo	92	2.2
10-3-3	6.6×10^{-3}	E	86	1.5
10-3-4A	8.8×10^{-3}	G	86	3.1×10^{-1}
9-5-1A	6.2×10^{-3}	E	84	1.3×10^{-1}

* All conductivity cells in this table were calibrated by the geometrical technique.

** Persistance Measuring Method:

K-1   E-External Calibrator

Vo   Vo-Constant transfer function $\frac{RV^0}{\sqrt{VI}}$

R-Vo   G-Graphical (R vs. Vo at fixed frequency)

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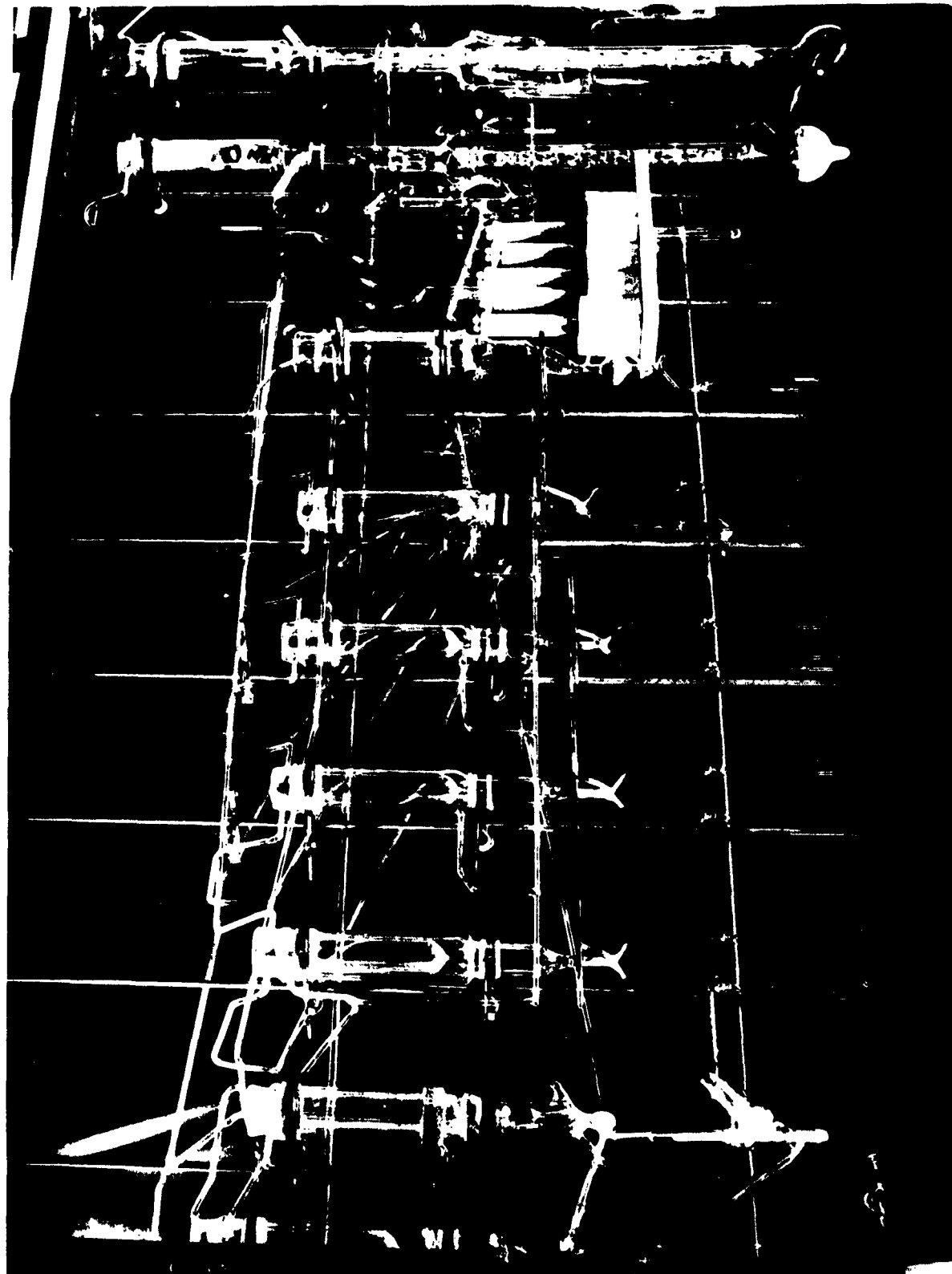
ACKNOWLEDGMENTS

FIGURE 1

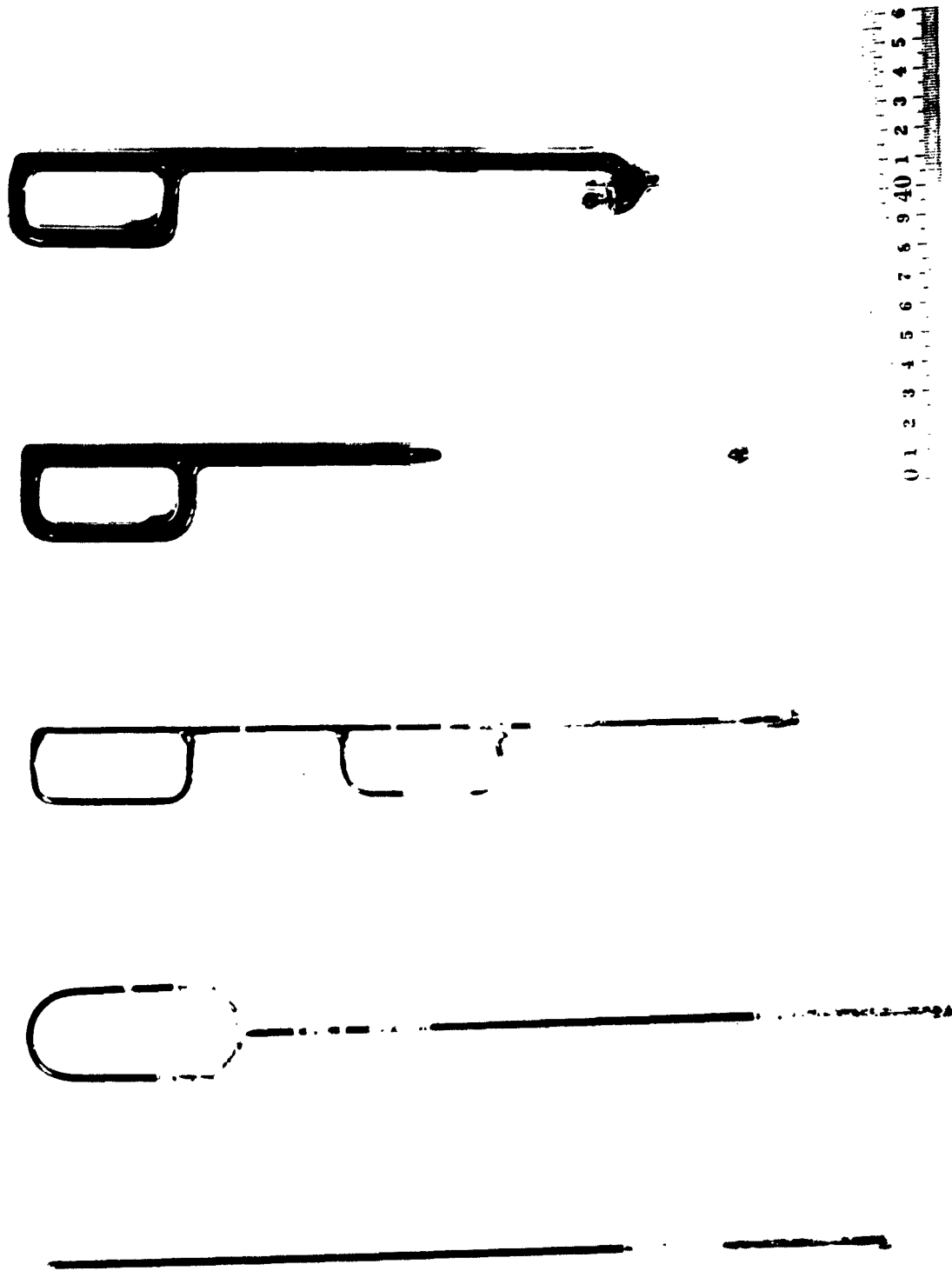Ammonia Purification and Sample Preparation System

FIGURE 2 - LIFETIME AND CONDUCTIVITY CELLS

The first cell on the left is a heavy wall capillary used for visually observing lifetimes at elevated temperature. The second cell is a capillary conductivity cell allowing visual inspection. The third cell was designed for measuring conductivities at several positions along the cell to study uniformity of conductivity when using the bomb. The fourth cell is a large-bore conductivity cell used in the bomb. In the last cell, a breakseal is built on for purposes of gas analysis.

FIGURE 3 - THE HIGH TEMPERATURE CONDUCTIVITY MEASURING SYSTEM

The conductivity cell is inside the bomb located on the rotary table at the left of the safety shield. Rotation of the bomb table, heating, and measurements are all controlled from the right.
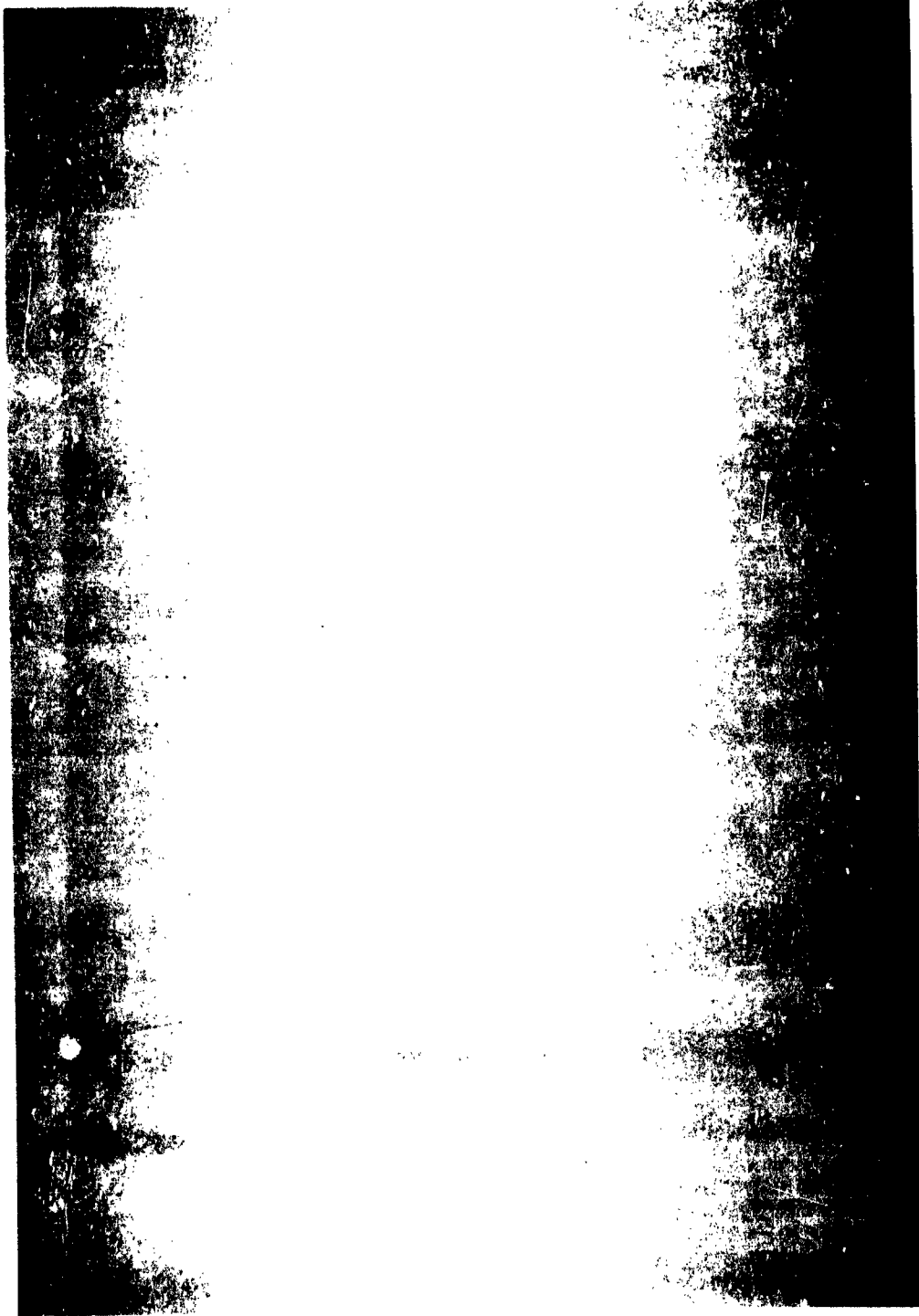
FIGURE 4 - CONDUCTIVITY ASSEMBLY INSIDE BOMB
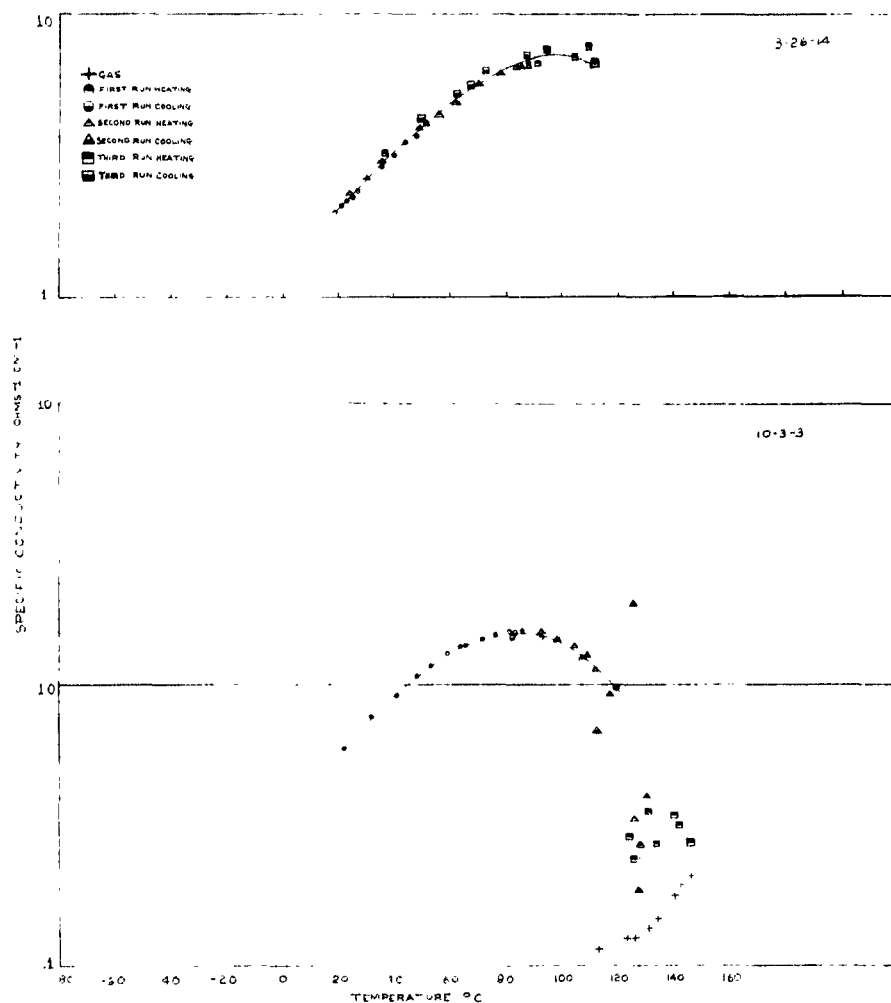
FIGURE 5 - REPRODUCIBILITY

Two test runs are shown, each of which was heated three separate times. There is no evidence of decomposition within experimental error for either run.
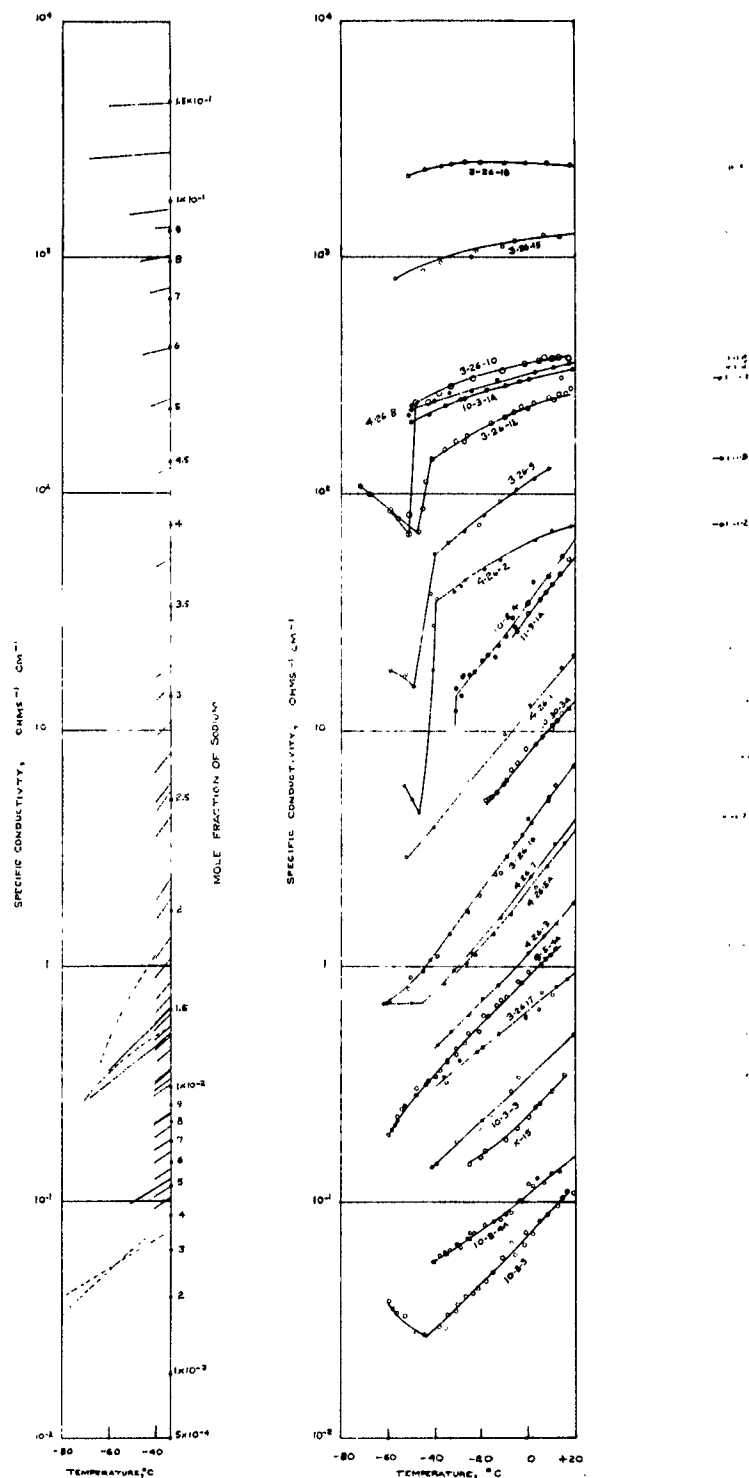
FIGURE 6 - ELECTRICAL CONDUCTIVITY TEMPERATURE DATA BELOW ROOM TEMPERATURE

The solid lines on the figure on the left are from the data of Kraus and Lucasse (11), the dash lines being from Mabauer(19). The concentration scale, mol fractions of sodium is from the data of Kraus(5) and Kraus and Lucasse(6). In the figure on the right, our conductivity temperature data below room temperature is displayed.
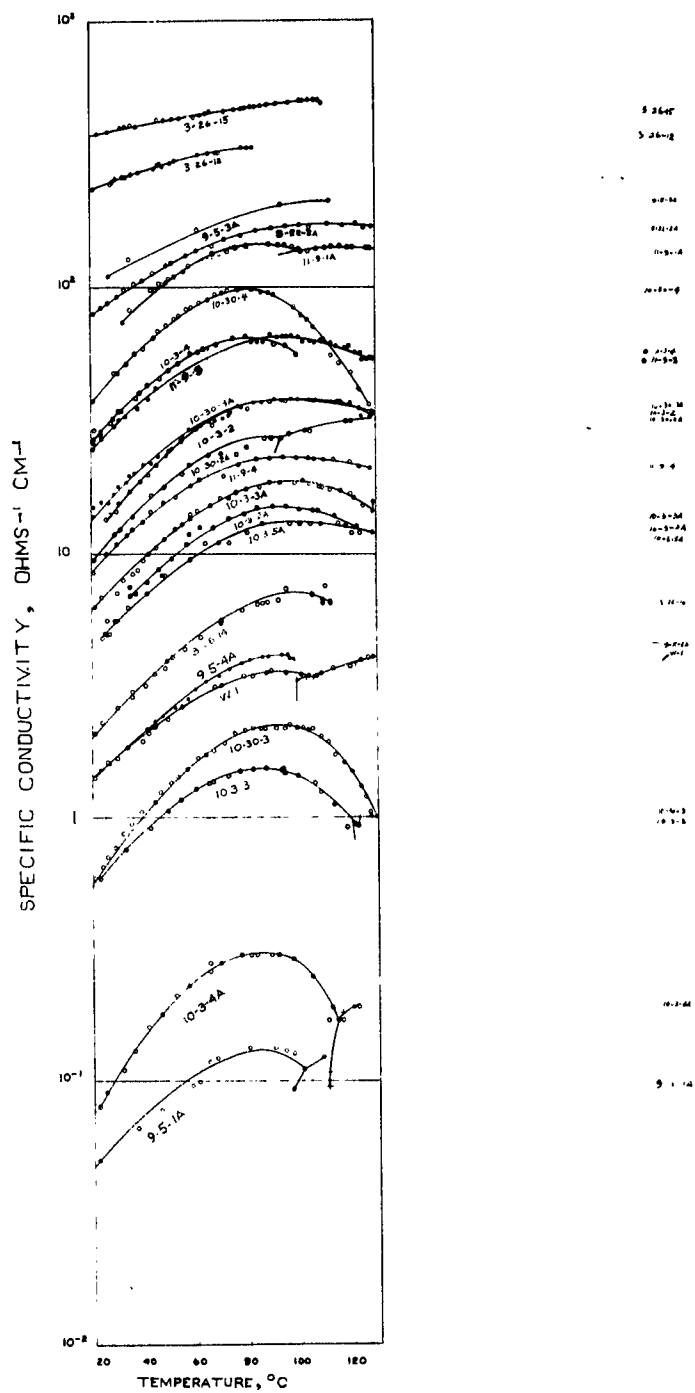
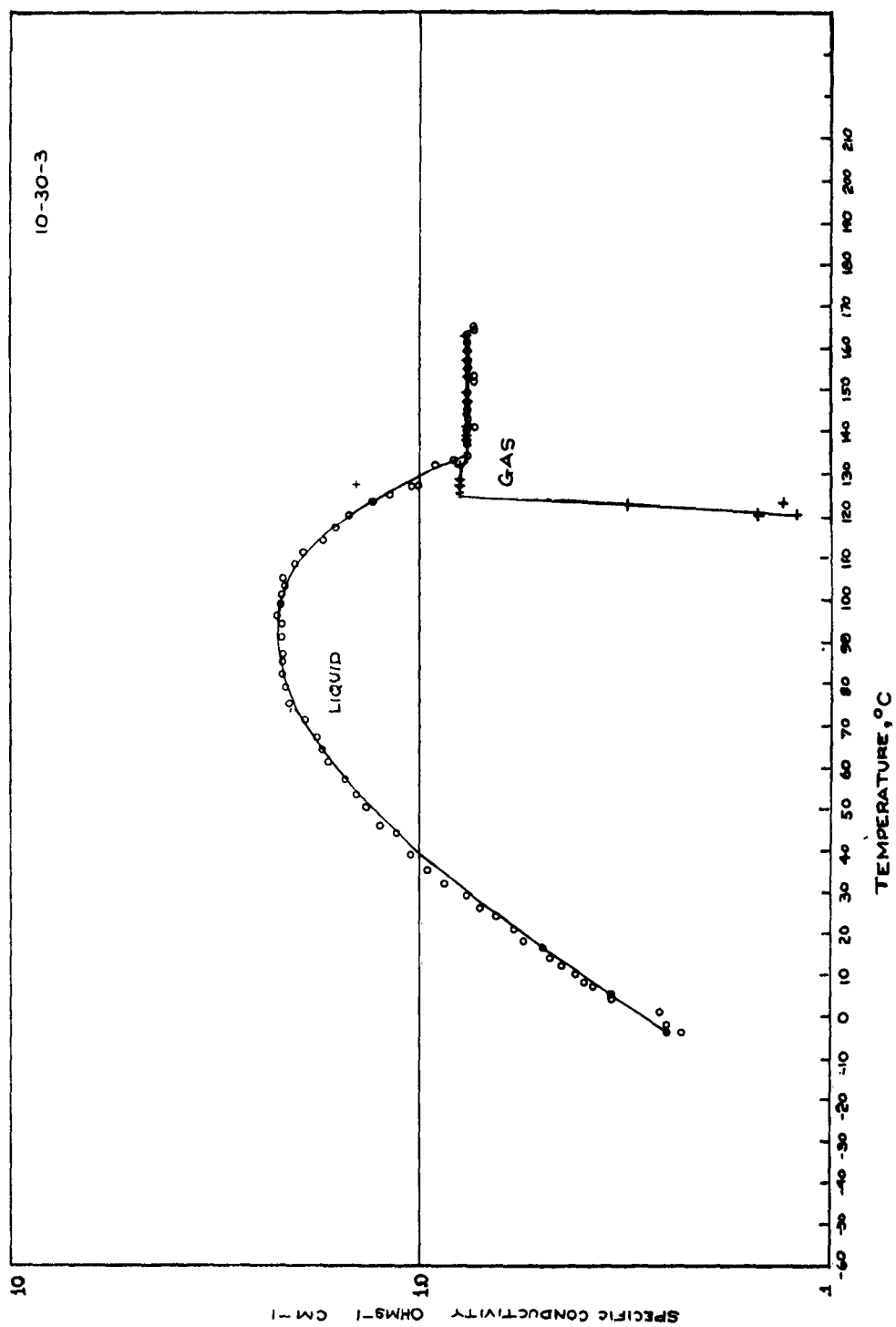FIGURE 7 - ELECTRICAL CONDUCTIVITIES - TEMPERATURE DATA TO 130°C

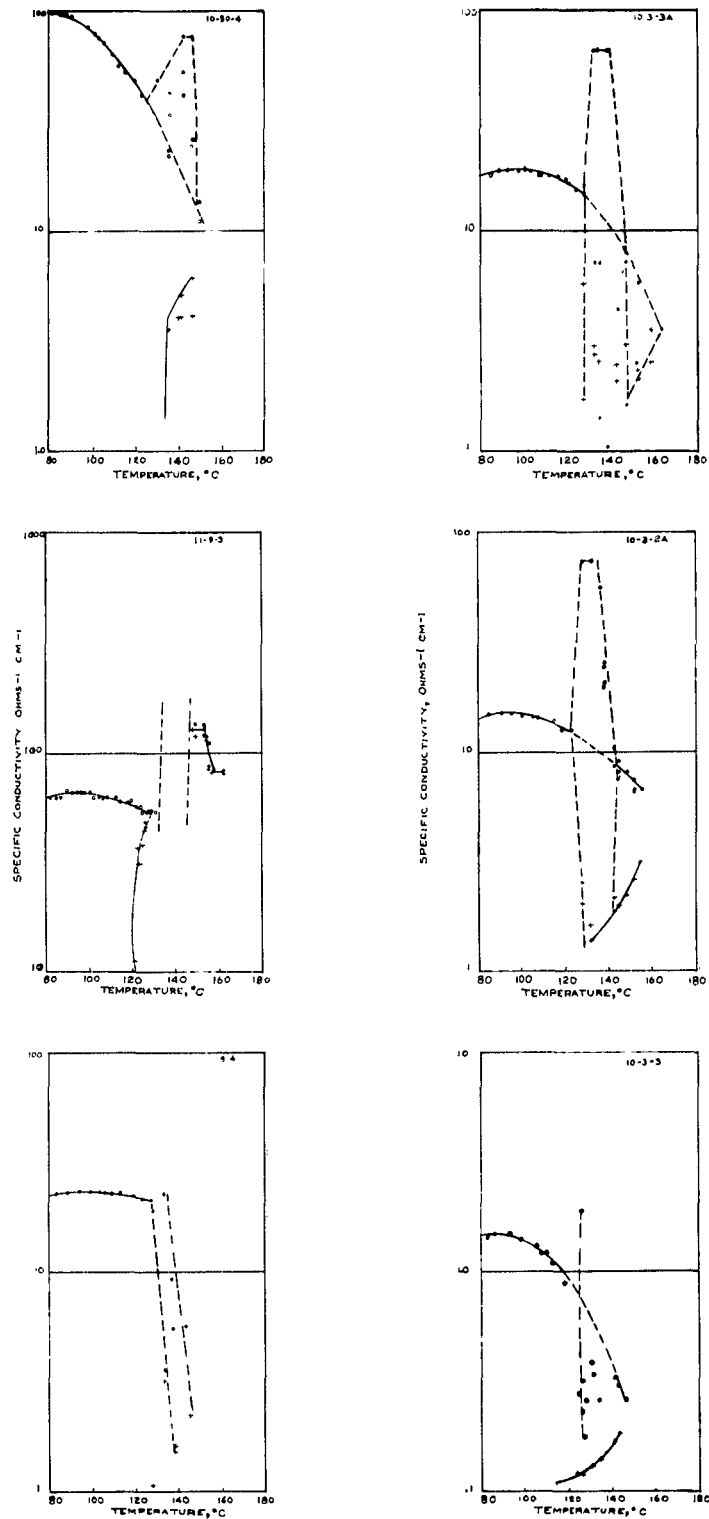FIGURE 8 - CONDUCTIVITY TEMPERATURE DATA EXTENDING INTO THE GAS PHASE REGION

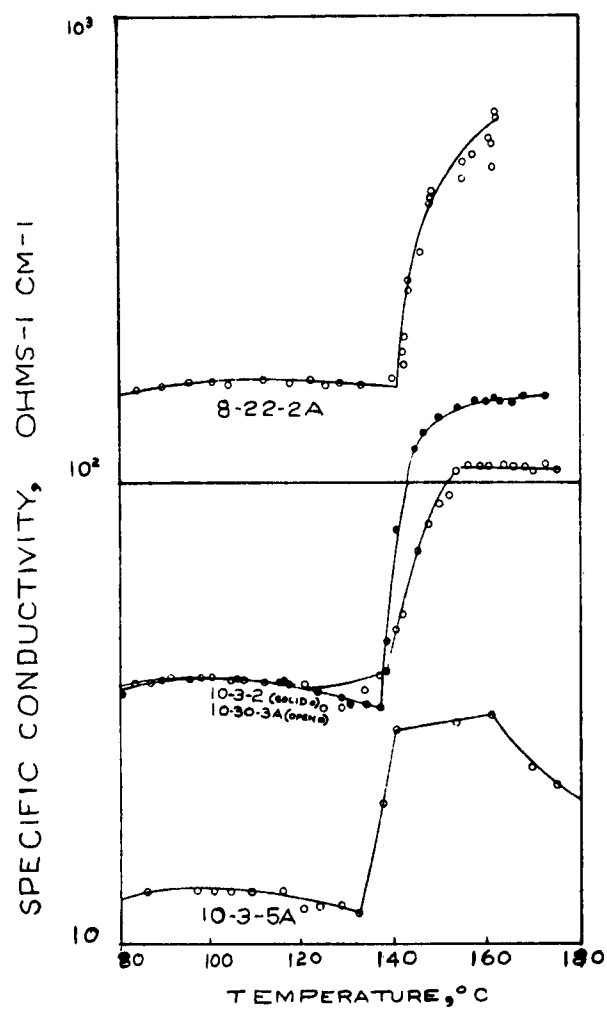FIGURE 9 - TURBULENCE IN THE NEIGHBORHOOD OF THE CRITICAL REGION

FIGURE 10 - CONDUCTIVITIES OF CONCENTRATED SOLUTIONS AT ELEVATED TEMPERATURES